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### COMPLETE SPECIFICATION.

#### Treatment of Cellulosic Fabrics.

- We, TOYO SPINNING COMPANY LIMITED, a Corporation of Japan, of 8, Dojima-Hamadori 2-chome, Kita-ku, Osaka, Japan, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—
- The present invention relates to the treatment of fabrics containing cellulosic fibres (natural or regenerated) to reduce their tendency to crease and wrinkle both in the wet and dry state or to enhance the dry and wet resilience or crease resistance of the fabrics without adversely affecting desirable properties and characteristics, particularly the wet and dry strength, of the fabrics.
- It is well known that when a cellulosic fabric is impregnated with formaldehyde together with a strong acid and subjected a cross-linking reaction, the tendency of the fabric to wet creasing may be reduced. This treatment, however, is not satisfactory to produce real "wash and wear" fabric because it fails to provide the fabric with a sufficient dry crease-resistance. A still further drawback of the conventional formaldehyde-strong acid treatment wherein formaldehyde and the strong acid are simultaneously applied in the liquid phase to a fabric is that the satisfactory wet crease-resistance is obtained only with a considerable sacrifice of mechanical strength, such as tearing strength and tensile strength, of the fabric.
- To improve the dry crease-resistance it has been proposed to treat a cellulosic fabric with a resinous substance (e.g. aminoplast resin) after the fabric has been subjected to the conventional formaldehyde-strong acid treatment. However, such additional treatment would be time-consuming and require additional cost. Furthermore, the desired mechanical strength of the fabric is further reduced by such additional treatment. A further drawback of the conventional resin treatment is in the fact that the resin is gradually washed away from the fabric by repeated laundering so that it is impossible for the fabric to retain the desired wash and wear performance for a prolonged period of time.
- In accordance with the present invention there is provided a process of treating a cellulosic fibre-containing fabric to improve its wet and dry resilience, which comprises impregnating the fabric with an aqueous liquid containing an aldehyde, or a substance which is capable of liberating an aldehyde, in the absence of an acid catalyst, and then contacting the fabric with gaseous hydrogen chloride, sulphur trioxide or sulphur dioxide, at a temperature below 50° C., while maintaining the moisture content of the fabric from 3 to 20% (preferably between 6 and 15%) by weight.
- The process of this invention is applicable to fabrics containing cellulosic fibres (either natural or artificial) alone or as mixtures of two or more in various proportions or as mixtures with other fibres. As natural cellulosic fibres, cotton, linen and hemp, may be exemplified, and among artificial cellulosic fibres are, for example, viscose rayon, cupra ammonium rayon and other regenerated cellulosic fibres. Other fibres which may be used with one or more of the above mentioned cellulosic fibres are, for example, cellulose acetate, polyamide, polyester, polyacrylonitrile, polyolefine, polyvinyl chloride, polyvinylidene chloride and polyvinyl alcohol

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fibres. If desired the fibres may be those graft-copolymerized with copolymerizable vinyl monomers. The fabric may be knit, woven or non-woven, or be any otherwise constructed fabric.

In carrying out the method of this invention, a fabric is first impregnated with an aqueous liquid containing an aldehyde or a substance which is capable of liberating an aldehyde under the treatment conditions, such as paraformaldehyde, methylol hydantoin, glyoxal, glutar aldehyde, acetal and hexamethylene tetramine. However formaldehyde is most preferred in this invention.

An aldehyde (e.g. formaldehyde or glyoxal) or a substance capable of liberating an aldehyde is applied to a fabric in the form of aqueous solution or liquid in any suitable manner such as by spraying the liquid on the fabric or immersing the fabric in the liquid followed by squeezing. In any event the fabric to be subjected to the subsequent gaseous acid treatment should contain 1 to 25%, preferably 4 to 15%, by weight of an aldehyde (as available component) or an aldehyde-liberating compound (as available aldehyde) based on the weight of the fabric. Generally, an aqueous liquid containing from 1 to 50% (usually 5 to 20%) by weight of an aldehyde (as available component) or containing an aldehyde-liberating substance in such an amount that it would liberate an aldehyde in a concentration of from 1 to 50% (usually 5 to 20%) by weight of the liquid is employed. The liquid is applied to the fabric by spraying or dipping so that the fabric has a wet pick-up of 50 to 100% (usually 60 to 80%) by weight. If necessary, the fabric is squeezed to attain the pick-up recited above.

It is preferable that the solution of an aldehyde or an aldehyde-liberating substance contains a formaldehyde (or other aldehyde) catcher. The term "catcher" as used herein is intended to mean a substance which is capable of holding or retaining formaldehyde (or other aldehyde) during the drying and subsequent acid treatment to prevent the dissipation of the aldehyde. Examples of such aldehyde catchers are polyacrylates such as polyethylacrylate, polybutylacrylate, polyethyl-butylacrylate and polyhydroxyethylacrylate, and cellulose ethers such as methyl cellulose, ethyl cellulose and hydroxyethyl cellulose. Usually these polyacrylates and cellulose ethers are commercially available as an aqueous solution or emulsion having a solid content of 20 to 40% by weight. In carrying out the invention, such an emulsion or solution is added to the aqueous liquid containing an aldehyde or an aldehyde-liberating substance so as to be in a concentration of from 1 to 10% (as such emulsion or solution) by weight.

The solution may also contain a small amount of suitable blueing agent, brightening agent or softener (lubricant). Particularly a softener or lubricant will affect the wash and wear properties and strength of the fabric resulting from the treatment according to this invention. For the purpose of this invention, preferable softeners or lubricants are polyolefine type softeners such as polyethylene or polypropylene, and silicone type softeners such as those commercially available under the name "Noran Silicone Softener". These softeners will impart to the fabric a desirable softness, good wash and wear properties and desired balance of dry and wet resilience and strength of the fabric. It is most preferable to employ both a polyolefine type softener and a silicone type softener. The amount of the softener or lubricant is not critical but generally a satisfactory result is obtained when it is added to the aldehyde (or aldehyde-liberating substance) solution in a concentration of from 1% to 6% by weight.

After impregnation with the liquid containing an aldehyde or an aldehyde-liberating substance, the fabric is contacted with gaseous hydrogen chloride, sulphur trioxide or sulphur dioxide and subjected to an acetalizing or cross-linking reaction between the cellulose and aldehyde. As mentioned before the moisture (calculated as water) content of the fabric during the contact with gaseous hydrogen chloride, sulphur trioxide or sulphur dioxide and the reaction should be so controlled as to be between 3 and 20% by weight based on the fabric weight. Therefore, it is usually necessary to dry the fabric before the acid treatment so that the fabric, when entering the gaseous acid-treating stage, has a moisture content within the required range, namely 3 to 20% (preferably 6 to 15%). The fabric may be dried in any suitable manner at a temperature from room temperature up to 120° C.

The fabric with the above mentioned moisture content is then contacted with the gaseous acid (catalyst for the reaction between the aldehyde and cellulose). This is accomplished by contacting or exposing the fabric to an atmosphere containing the gaseous acidic substance. The atmosphere to be contacted with the fabric should contain 1 to 100% by volume, preferably 5 to 40% by volume, of gaseous hydrogen chloride, sulphur trioxide or sulphur dioxide and be maintained at a temperature below 50° C., preferably between 20° C. and 50° C. By this contact, the acidic substance or catalyst is applied to the fabric because the gas is dispersed in and absorbed by the fabric.

To contact the fabric with the gaseous acidic substance any suitable means may be employed. For example, an open vessel containing hydrochloric acid in a desired

concentration is placed at the bottom of a closed chamber and the said container is heated so that gaseous hydrogen chloride vaporized from the container will fill the interior of the chamber through which the fabric may be passed. Alternatively, gaseous hydrogen chloride, sulphur trioxide or sulphur dioxide may be separately prepared and continuously introduced into a closed chamber through which the fabric is passed. If desired, it is possible to directly blow the acidic gas against the travelling fabric. Usually the fabric is continuously passed in one direction and the gaseous acidic substance is applied to the fabric at a predetermined point during its passage. Alternatively, the fabric is placed in a closed chamber (containing the gaseous acidic substance) rolled up on a rotatable roller. A second rotatable roller is arranged within the same chamber and spaced from the first roller. Within the chamber the roller is driven so that the fabric roll on the first roller is continuously unwound therefrom and rewound up on the second roller. When the fabric is completely wound up on the second roller, the driving direction is reversed so that the fabric is unwound from the second roller and rewound up around the first roller. During the movement from the first roller to the second roller or vice versa the fabric is contacted with or exposed to the acidic gas. This unwinding and rewinding is repeated until the fabric has contacted the treating gas for a desired period of time. In still another embodiment, the fabric is contacted with a continuously travelling porous belt impregnated with hydrochloric acid of a desired concentration so that during the contact with the belt the fabric is contacted with hydrogen chloride gas developed from the hydrochloric acid within the belt. In this case, care should be taken that the fabric is not directly contacted with the hydrochloric acid liquid contained in the belt.

The contact of the fabric with gaseous hydrogen chloride, sulphur dioxide or sulphur trioxide should be effected at a temperature below 50° C., preferably between 20° C. and 50° C. The time during which the said contact is carried out varies widely depending upon the temperature, the particular acid employed and the concentration of acid in the atmosphere applied to the fabric. Hydrogen chloride and sulphur trioxide are more reactive and therefore require shorter treating times and/or lower temperatures than sulphur dioxide. When the acid concentration in the atmosphere to be applied to the fabric is higher, a shorter time and/or lower temperature may be employed. However, the contact time should be such that the fabric absorbs the acidic catalyst in an amount sufficient to cause the desired acetalization or cross-linking reaction. As men-

tioned above the time for contacting the fabric with the gaseous acidic catalyst varies over a wide range, namely only one or a few seconds to 90 minutes or more, depending largely upon the concentration of the gas and the temperature at which the contact is carried out.

Upon contact of the fabric with the gaseous acid catalyst the reaction between the aldehyde and cellulose commences. The desired reaction may be completed while the fabric is contacted with the gas. Alternatively, it is possible to contact the fabric with the gas until a desired amount of the acidic catalyst is applied to or absorbed by the fabric at a temperature below 50° C. and then leave the fabric to stand in an acid-free atmosphere at a temperature below 50° C. until the reaction proceeds to the desired extent, if the reaction has not proceeded to the desired extent during the contact of the fabric with the gas.

By way of examples, when a cotton fabric is immersed in an aqueous solution containing 35% by weight of formaldehyde (38%), squeezed to a wet pick-up of 70% and then dried to a moisture content of 10% by weight, a satisfactory result is obtained if the dried fabric is passed through a closed chamber containing hydrogen chloride gas at the following concentration and temperature for the following reaction time:—

Temperature (° C.)	HCl gas conc. (vol. %)	Reaction Time (seconds)	
20	10	180	100
20	40	60	
30	10	90	
30	40	15	105
40	10	30	
40	40	5	
40	5	120	

In still another example the same dried fabric was passed through a chamber wherein a gas containing 5% by volume of hydrogen chloride gas was blown against the fabric at 30° C. for 10 minutes. Immediately thereafter the fabric was left to stand in an acid-free atmosphere at 30° C. for 90 seconds to obtain a result substantially the same as that obtained in the examples given in the above table.

Thus, the time for contacting the fabric with the gaseous acidic catalyst and whether or not the fabric should be left standing after the said contact vary widely under the particular circumstance involved but should be easily determined by those skilled in the art.

The gas to be applied to the fabric may contain, in addition to the gaseous acid, other

- gaseous components such as air, nitrogen gas and moisture (water) which do not affect the reaction involved. The moisture condition in the treating atmosphere may be neglected in connection with the moisture content of the fabric mentioned above.
- Throughout the handling and treatment of the fabric, inclusive squeezing, drying, acid treatment and the subsequent reaction (if any), the fabric should be kept at its open width because the creases or wrinkles formed on the fabric during the process are difficult to remove or recover and lead to critical loss in the marketability of the product.
- Immediately after the acid treatment or the subsequent reaction (if any), the fabric is washed to remove any acid and free material remaining on the fabric. The washed fabric is then dried.
- A cellulosic fabric may be subjected to the method of this invention without any pretreatment of the fabric. However, it is preferable to employ a preliminarily mercerized fabric in this invention, because using the method of this invention it is easier to impart desirable wash and wear to a mercerized fabric. Mercerization is well known in the art and no detailed explanation thereabout appears to be necessary.
- It has further been found that when the fabric is subjected to alkylation or substituted-alkylation before carrying out the method of this invention, improved characteristics of wash and wear properties and improved balance between the wet and dry resilience and strength of the fabric are obtained according to this invention. The alkylation or substituted-alkylation may be carried out in a conventional manner by treating the fabric with an alkylating (or substituted-alkylating) agent in an alkaline medium. Thus, for example, a dialkyl sulphate, e.g. dimethyl sulphate or diethyl sulphate; an alkyl or substituted alkyl halide, e.g. butyl chloride, ethyl chloride, butyl bromide or ethyl bromide; ethylene chlorohydrin, ethylene bromohydrin, chloroacetic acid or bromoacetic acid, may be used as an alkylating or substituted-alkylating agent.
- When hydroxyalkylation is desired ethylene oxide or propylene oxide may be employed. In carrying out the alkylation or substituted-alkylation, the fabric is impregnated with an aqueous solution (5% by weight or more in concentration) of the alkylating (or substituted alkylating) agent and squeezed. The fabric is further impregnated with a strong (10% by weight or more) aqueous alkali solution, for example, of sodium hydroxide or potassium hydroxide, and then squeezed. If the alkylating or substituted-alkylating agent is insoluble in water it may be applied to the fabric in the form of an emulsion. If the alkylating or substituted-alkylating agent is reactive with water it may be applied to the fabric in the form of a solution in an organic solvent, for example, alcohol or benzene. The alkylation or substituted-alkylation may be carried out on a conventional mercerizing machine. It is preferable that the degree of alkylation or substituted-alkylation is such that 2 to 5 mols of alkyl or substituted alkyl groups are introduced per 100 glucose units constituting the fabric fibres.
- The invention will be further illustrated by reference to the following examples. In these examples all percentages are by weight and various properties of the fabrics have been determined by the following methods:—
- |                          |   |    |
|--------------------------|---|----|
| Tensile strength         | : ASTM D39—49 Grab method, in respect of warps.   | 70 |
| Wet resilience           | : By the method described in American Dyestuff Reporter, Vol. 45, P. 472 (1956), and indicated by the sum of values for fillings and warps. | 75 |
| Dry resilience           | : ASTM-D-1295-53T; indicated by the sum of values for fillings and warps.   | 80 |
| Wash and wear properties | : Determined by comparing with Monsanto Plastic Model.  | 85 |
| Tear strength            | : ASTM-D-1424-56T (Elmendorf method).   | 90 |
| Laundry                  | : By AATCC Tentative Test Method 88-1960-4a.  | 95 |
- EXAMPLE 1.**
- A sample of mercerized cotton poplin was immersed in a bath of the following composition, squeezed to a wet pick up of 65% and dried at 100° C. to reduce the moisture content to 8% by weight.
- |                             |     |      |     |
|-----------------------------|-----|------|-----|
| Formaldehyde (37%)          | ... | 15%  |     |
| Polyethylene emulsion (20%) | ... | 2%   |     |
| Noran Silicone Softener     | ... | 2%   |     |
| Water to                    | ... | 100% | 110 |
- The dried cotton fabric was then passed through a closed chamber at 30° C. for 12 minutes, while supplying the chamber with hydrogen chloride gas so that the concentration of hydrogen chloride gas within the chamber is maintained at about 3% by volume. During the passage through the chamber the fabric was contacted with said gaseous hydrochloric acid therein. Immediately thereafter, the fabric was washed with water and dried without any tension. The fabric was tested and the results are as follows:—
- |                        |     |             |     |
|------------------------|-----|-------------|-----|
| Wet resilience         | ... | 283 degrees |     |
| Dry resilience         | ... | 261 degrees | 125 |
| Tensile strength       | ... | 15.1 kg.    |     |
| Wash and wear property |     | 4.9         |     |

The resulting fabric had a neat appearance and a very soft hand, and there was no increase in the weight. The wet and dry resilience and the wash and wear properties of the fabric were improved remarkably by the above treatment, and there was no noticeable reduction in the tensile strength or tearing strength. There was no appreciable reduction in the wet and dry resilience of the fabric even after ten cycles of laundering.

#### EXAMPLE 2.

The procedure of Example 1 was repeated except that the fabric, after the immersion in the formaldehyde solution and the subsequent drying, was passed through a cham-

ber maintained at 20° C. for 15 minutes. Within the chamber and at the bottom thereof an open container had been placed containing 36% hydrochloric acid so that the chamber contained about 6% by volume of hydrogen chloride gas during the treatment of the fabric. Immediately after this hydrogen chloride treatment, the fabric was washed with water and dried. The treated fabric was tested and the results are given in the following table. In this table are also given the corresponding data in respect of a conventional resin finished fabric and also in respect of a conventional formaldehyde-treated fabric for comparison.

Sample	Resilience (degrees)		Tensile strength (kg.)	Wash and wear properties	
	dry	wet		After 1 cycle of laundering	After 10 cycles of laundering
Untreated	—	—	28.8	—	—
A	252	278	15.6	4.8	4.7
B	230—250	210—230	17.0—18.5	3.3	3.0
C	200—220	230—250	17.5—19.0	2.8	—

A: The fabric treated according to Example 2.

B: The resin finished fabric. The fabric was immersed in an aqueous bath containing 10% of triazone type resin precondensate (50%) 2% of zinc nitrate (40%) and 2% of polyethylene emulsion (20%). The fabric was squeezed to a wet pick-up of 65% and dried at 125° C. for 2 minutes. The fabric was then subjected to baking at 160° C. for 3 minutes.

C: The conventional formaldehyde-treated fabric. The fabric was immersed in an aqueous bath containing 10% of formaldehyde (37%), 2% of oxalic acid (crystal) and 2% of polyethylene emulsion (20%). The fabric was squeezed to a wet pick-up of 65% and dried at 125° C. for 3 minutes. Then the fabric was dried at 160° C. for 3 minutes.

#### EXAMPLE 3.

A poplin of viscose rayon staple fibres graft copolymerized with ethyl acetate (the rate of graft: 35% by weight) was immersed in an aqueous solution containing 30% of formaldehyde (37%) and 3% of ethyl cellulose (solid content 20%). The fabric was squeezed to a wet pick of 65% and dried at 110° C. to reduce the moisture content to

10%. The dried fabric was then passed through a closed chamber (25° C.) in which the air had been substantially replaced by hydrogen chloride gas so that the chamber contained about 6% (by volume) of hydrogen chloride gas. After passing through the chamber for 10 minutes, the fabric was washed with water and dried. The fabric was tested and the results were as follows:

	Resilience (degrees)		Tensile strength	Wash and wear properties
	Dry	Wet		
Before treatment (without grafting)	...	...	23.2 kg.	—
After treatment	...	...	18.1 kg.	4.6

#### EXAMPLE 4.

A mercerized cotton poplin was immersed in an aqueous solution containing 13% of hexamethylene tetramine, 2% of Noran Silicone Softener (a silicone type softener), 4%

of polyethylene emulsion (solid content 20%) and 0.01% of a penetrating agent (polyoxyethylene glycol ether non-ionic surface active agent). The fabric was then squeezed to a wet pick-up of 70% and dried at 110° C. to

reduce the moisture content to 10%. Then the fabric was contacted for 20 minutes with a polystyrene sponge sheet impregnated with 36% hydrochloric acid and heated at 48° C.

- 5 During this contact, the fabric was exposed to a gas containing 1% (by volume) of hydrogen chloride gas. Immediately after this hydrochloric treatment the fabric was washed and dried, and subjected to testing.
- 10 The results were as follows:—

	Treated	Untreated
Tensile strength	25.3 kg.	30 kg.
Tear strength	1459 g.	1000 g.
Resilience:		
15 Dry	215 degrees	—
Wet	221 degrees	—
Wash and wear performance	3.5	

#### EXAMPLE 5.

- 20 A mercerized cotton poplin was immersed in an aqueous liquid containing 35% of formaldehyde (37%), 2% of polyethylene emulsion (solid content 20%) and 2% of polyethylbutylacrylate (solid 30%). Then the fabric was squeezed to a wet pick-up of 65% and dried to a moisture content of 10%. The fabric was wound up on a roller and placed within an autoclave in which was arranged a second winding roller spaced from the first roller. The air in the autoclave was replaced by sulphur dioxide so that the interior volume of the autoclave contained about 10% by volume of sulphur dioxide at 48° C.
- 30 The rolled fabric was unwound from the first roller and rewound on the second roller and then vice versa. This unwinding and re-winding was repeated until the fabric had contacted the sulphur dioxide gas atmosphere for 20 minutes. Then the fabric was washed with water and dried. The fabric was tested and the results were as follows:—

Tensile strength ...	13.1 kg.
Tear strength ...	278 g.

#### Resilience:

Dry ...	263 degrees	45
Wet ...	275 degrees	
Wash and wear properties	4.1	

#### EXAMPLE 6.

A mercerized cotton poplin was immersed in an aqueous liquid containing 35% of formaldehyde (37%), 2% of polyethylene emulsion (solid 20%) and 2% of polyethylbutyl acrylate (solid 30%). Then the fabric was squeezed to a wet pick-up of 65% and dried at a temperature of 100° C. to a moisture content of 10%. The fabric was contacted with a continuously travelling polystyrene sponge containing 36% hydrochloric acid for 15 minutes at 40° C. so that the fabric was exposed to an atmosphere containing about 3% by volume of gaseous hydrogen chloride. After washing and drying the fabric was tested. This fabric had a wash and wear performance of 4.5.

#### EXAMPLE 7.

A mercerized cotton poplin was immersed in an aqueous solution containing 10% by weight of monochloroacetic acid, squeezed to a wet pick-up of 65%, then immersed in 30% aqueous solution of sodium hydroxide and squeezed to a wet pick-up of 70%, and the reaction was allowed to proceed for 2 minutes while holding the edges of the fabric to keep the same open. Then the fabric was washed with water, neutralized, again washed and dried.

The carboxymethylated poplin was immersed in an aqueous liquid containing 30% of formaldehyde (37%) and 4% of polyethylene emulsion (solid 20%). Then the fabric was squeezed to a wet pick-up of 65% and dried to a moisture content of 10%. The fabric was then passed through a closed chamber filled with an atmosphere containing 80% (by volume) of gaseous hydrogen chloride, at 35° C. for 5 seconds. For comparison, the same procedure was repeated with the same mercerized cotton poplin but not carboxymethylated. The test results for these fabrics after washing and drying were as follows:—

	Resilience		Tensile strength	Wash and wear properties
	Dry	Wet		
Untreated ...	—	—	28.0 kg.	—
95 Carboxymethylation + formaldehyde treatment ...	245	280	14.5 kg.	5.0
Formaldehyde treatment only ...	240	270	13.5 kg.	4.7

#### EXAMPLE 8.

- A mercerized cotton poplin was immersed in the aqueous formaldehyde solution of Example 6, squeezed to a wet pick-up of 65%

and dried at 100° C. to a moisture content of 10%. Then the fabric was passed through a closed chamber of an atmosphere containing 10% (by volume) of hydrogen chloride

gas at 40° C. for 30 seconds. For comparison the same procedure was repeated with using the same mercerized cotton poplin but

carboxymethylated as in Example 7. The test results for these fabrics are as follows:—

					Resilience Dry	Wet	Tensile strength	Wash and wear properties
10	Carboxymethylation +							
	formaldehyde treatment ... ..				270	290	14.1 kg.	4.8
	Formaldehyde treatment only ... ..				240	270	13.7 kg.	4.5
	Untreated ... ..				—	—	28.0 kg.	—

#### EXAMPLE 9.

The procedure of Example 7 was repeated except that a 15% aqueous solution of ethylenechlorohydrin was employed instead of the monochloroacetic acid solution. The tests on this fabric disclosed that it has excellent wash and wear properties and excellent balance between resilience and strength.

liquid containing 20% of formaldehyde (35%), 4% of polyethylene emulsion (solid content (20%) and 2% of polyethyl-butyl-acrylate (solid content 30%). Then the fabric was squeezed to a wet pick-up of 65% and dried at 100° C. to a moisture content of 12%. The fabric was passed through a closed chamber of an atmosphere containing 5% (by volume) of hydrogen chloride gas at 48° C. for 30 seconds. Then the fabric was washed and dried, and tested. The test results were as follows:—

#### EXAMPLE 10.

A satin cloth composed of regenerated cellulose fibres having a high degree of polymerisation was immersed in an aqueous

						Resilience Dry	Wet	Tensile strength	Wash and wear properties
40	Example 10	...	...	...	...	267	286	11.kg.	4.5
	Untreated	...	...	...	...	—	—	20 kg.	—

#### EXAMPLE 11.

A cotton poplin was immersed in an aqueous bath containing 35% of formaldehyde (38%), 4% of polyethylene emulsion (solid 20%) and 2% of Noran silicone softener. The fabric was squeezed to a wet pick-up of 70% and dried to a moisture content of 8%. The dried fabric was passed through a closed chamber maintained at 30° C. for 90 seconds while feeding hydrogen chloride gas to the chamber so that the latter contained about 12% (by volume) of hydrogen chloride gas during the treatment. Immediately after this treatment the fabric was washed and dried. The test results were as follows:—

45	Tensile strength	...	...	15.0 kg.
	Tear strength	...	...	700 g.
50	Dry resilience	...	...	270 degrees
	Wet resilience	...	...	290 degrees
60	Wash and wear properties	...	...	4.7

tent of 7%. Then the fabric was passed for 25 seconds through a closed chamber maintained at 40° C. and containing 14% by volume of hydrogen chloride gas. After the treatment, the fabric was washed and dried. The properties of this fabric were as follows:

Tensile strength	...	...	15.5 kg.
Tear strength	...	...	700 g.
Dry resilience	...	...	270 degrees
Wet resilience	...	...	300 degrees
Wash and wear properties	...	...	5.0

#### EXAMPLE 13.

A cotton poplin was immersed in the aqueous solution of Example 11, squeezed to a wet pick-up of 75% and dried to a moisture content of 7%. Then the fabric was passed for 20 seconds through a closed chamber maintained at 30° C. and containing 7% by volume of hydrogen chloride gas. After passing through the chamber, the fabric was left standing for 60 seconds in an atmospheric condition at 30° C. Then the fabric was washed and dried. The properties of the fabric were as follows:

EXAMPLE 12.  
A cotton poplin was impregnated with the solution of Example 11, squeezed to a wet pick-up of 75% and dried to a moisture con-

Tensile strength ... ..	15.0 kg.
Tear strength ... ..	700 g.
Dry resilience ... ..	270 degrees
Wet resilience ... ..	270 degrees

#### 5 EXAMPLE 14.

A cotton poplin was immersed in an aqueous liquid containing 10% of glyoxal, 4% of polyethylene emulsion (solid 20%) and 2% of Noran silicone softener. The fabric was squeezed to a wet pick-up of 75% and dried to a moisture content of 10%. Then the fabric was passed for 40 seconds through a closed chamber maintained at 40° C. and containing 10% (by volume) of hydrogen chloride gas. Immediately after this acid treatment the fabric was washed and dried. The properties of the fabric after the treatment were as follows:—

Tensile strength ... ..	16.0 kg.
Tear strength ... ..	750 g.
Dry resilience ... ..	240 degrees
Wet resilience ... ..	285 degrees
Wash and wear properties	4.5

#### WHAT WE CLAIM IS:—

25 1. A process of treating a cellulosic fibre-containing fabric to improve its wet and dry resilience, which comprises impregnating the fabric with an aqueous liquid containing an aldehyde, or a substance which is capable of liberating an aldehyde, in the absence of an acid catalyst, and then contacting the fabric with gaseous hydrogen chloride, sulphur trioxide or sulphur dioxide, at a temperature below 50° C. while maintaining the moisture content of the fabric from 3 to 20% by weight.

40 2. A process according to Claim 1, wherein the moisture content of the fabric is maintained at between 6% and 15% by weight.

3. A process according to either of Claims 1 to 2 wherein the aldehyde or aldehyde-liberating substance is impregnated in the fabric in an amount of 1 to 25%, preferably 4 to 15%, by weight based on the weight of the fabric. 45

4. A process according to any one of the preceding claims, wherein the aqueous liquid containing the aldehyde or aldehyde-liberating substance also contains a polyolefine type softener or a silicone type softener. 50

5. A process according to any one of the preceding claims wherein the fabric is contacted with the gaseous acidic substance by exposing the fabric to an atmosphere containing 1% to 100% by volume of the gaseous acidic substance. 55

6. A process according to any one of the preceding claims, wherein the fabric is mercerized prior to said impregnation. 60

7. A process according to any one of the preceding claims, wherein the fabric is subjected to an alkylation or substituted-alkylation treatment prior to said impregnation.

8. A process according to any one of the preceding claims wherein the aldehyde is formaldehyde or glyoxal, or alternatively the aldehyde-liberating compound is hexamethylene tetramine. 65

9. A process for treating a cellulosic fibre-containing fabric to improve its wet and dry resilience according to Claim 1 and substantially as hereinbefore described with reference to the examples. 70

10. A cellulosic fibre-containing fabric whenever treated by a process according to any one of the preceding claims. 75

STEVENS, LANGNER, PARRY  
& ROLLINSON,  
Chartered Patent Agents,  
Agents for the Applicants.